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PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant : Shoji, et al.
Appl. No. : 10/642,952
Filed : August 18, 2003
For : PROCESS FOR PRODUCING
DIMETHYL ETHER
Examiner : Rosalynd Ann Keys
Group Art Unit : 1621

CERTIFICATE OF MAILING

I hereby certify that this correspondence and all marked attachments are being deposited with the United States Postal Service as first-class mail in an envelope addressed to: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450, on

(Date)

Katsuhiko Arai, Reg. No. 43,315

DECLARATION UNDER RULE 132

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

Dear Sir:

I, Satoshi Terai, do hereby declare as follows:

- 1 I am a co-inventor of the above-identified application.
- 2 The following experiments were conducted by me or under my supervision or control to show the relationship between Na₂O wt% of an activated alumina catalyst and the conversion rate of methanol to dimethyl ether.

3 Five (5) mL of a γ -alumina catalyst having a specific surface area (SA), a pore volume (PV), an average pore radius (R), and a Na₂O content shown in a table below (measured by the methods described on page 14 of the specification) were loaded in a stainless fixed bed reactor with an inner diameter of 16 mm equipped with an electric furnace around its periphery (no additional active component was added to the catalyst). Except for the γ -alumina catalysts having a Na₂O content of 0.20 wt% and 0.25 wt%, the Na₂O content of the γ -alumina catalyst was reduced by either selecting low-Na content raw materials or conducting acid washing. At a reaction temperature of 270°C, methanol was then fed at a GHSV of 1700 h⁻¹ and a pressure of 0 MPa-G (gauge pressure), while methanol was heated to its evaporating temperature at the reaction pressure in an upstream line of the reactor so that methanol was vaporized before



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entering the catalyst layer. During the process, a temperature of the catalyst layer was 270°C. A conversion ratio of methanol to DME was shown in the table under these conditions.

Table

Na2O [wt%]	Conversion @0MPa-G, 270°C [%]	PV [ml/g]	SA [m ² /g]	R [nm]
0.0015	78	0.59	256	4.6
0.0019	77	0.45	252	3.6
0.0022	77	0.71	258	5.6
0.020	76	0.37	115	6.5
0.038	70	0.74	200	7.4
0.080	60	0.47	171	5.5
0.20*	32	0.46	219	4.2
0.25*	23	0.35	117	6
0.25*	16	0.31	206	3

*: No Na2O reduction treatment was applied.

4 The above data are shown in graphs below.

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5 As shown in Fig. 1, the conversion ratio is highly correlated to the Na_2O content of the catalyst, and when the Na_2O content is about 0.07 wt% or less, the conversion ratio reaches about 70% or higher.

6 In contrast, the conversion ratio is not correlated to PV, SA, or R in the ranges used in these experiments as shown in Figs. 2-4. These figures also show that the Na_2O content is about 0.07 wt% or less, the conversion ratio reaches about 70% or higher.

7 I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true, and further that these statements were with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statement may jeopardize the validity of the application or any patent issuing thereon.

Respectfully submitted,

Dated: December 17, 2004.

By:

Satoshi Terai
Satoshi Terai

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